JC14 Rec'd PCT/PTO 16 JAN 2002

FORM PTO-1390 (REV 10-95) U.S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. §371	TAKIT 162
	U.S. APPLICATION NO. (If known, see 37 CFR §1.5) 10/031081

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/JP00/08326	27 NOVEMBER 2000	17 MAY 2000

TITLE OF INVENTION

METHOD OF DETERMINING STRUCTURE OF SOFT MATERIAL

APPLICANT(S) FOR DO/EO/US

TERASAKI, Osamu, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ■ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. §371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. §371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ■ A copy of the International Application as filed (35 U.S.C. §371(c)(2))
   - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   - b. ■ has been transmitted by the International Bureau.
   - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ■ A translation of the International Application into English (35 U.S.C. §371(c)(2)).
7. ■ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3))
   - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   - b. ☐ have been transmitted by the International Bureau.
   - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   - d. ■ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. §371(c)(3)).
9. ■ An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 C.F.R. §§1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
    ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

JC13 Rec'd PCT/PTO 16 JAN 2002

U.S. APPLICATION NO. (if known, see 37 CFR §1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
10/031081	PCT/JP00/08326	TAKIT 163

17. ☒ The following fees are submitted:		CALCULATIONS	PTO USE ONLY
BASIC NATIONAL FEE (37 CFR §1.492 (a) (1) - (5)):			
Search Report has been prepared by the EPO or JPO	$890.00		
International preliminary examination fee paid to USPTO (37 CFR §1.482)	$710.00		
No international preliminary examination fee paid to USPTO (37 CFR §1.482) but international search fee paid to USPTO (37 CFR §1.445(a)(2))	$740.00		
Neither international preliminary examination fee (37 CFR §1.482) nor international search fee (37 CFR §1.445(a)(2)) paid to USPTO	$1040.00		
International preliminary examination fee paid to USPTO (37 CFR §1.482) and all claims satisfied provisions of PCT Article 33(2)-(4)	$100.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =		$890.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 C.F.R. §1.492(e)).			

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	6 - 20 =	0	x $ 18.00	$0.00	
Independent claims	1 - 3 =	0	x $ 84.00	$0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ $ 280.00		
TOTAL OF ABOVE CALCULATIONS =				$890.00	
Reduction of 1/2 for filing by small entity, if applicable. A Verified Small Entity Statement must also be					
SUBTOTAL =				$890.00	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 C.F.R. §1.492(f)).					
TOTAL NATIONAL FEE =				$890.00	
Fee for recording the enclosed assignment (37 C.F.R. §1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. §§3.28, 3.31). $40.00 per property.					
TOTAL FEES ENCLOSED =				$890.00	
				Amount to be refunded:	
				charged:	

a. ■ A check in the amount of $890.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 13-3402 in the amount of $ ______ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ■ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3402. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 C.F.R. §§1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. §1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO: Customer Number 23,599

23599
PATENT TRADEMARK OFFICE

SIGNATURE

Anthony J. Zelano
NAME

27,969
REGISTRATION NUMBER

Filed: 16 JANUARY 2002
AJZ:kmo

10/031081
JC13 Rec'd PCT/PTO 16 JAN 2002

DESCRIPTION

METHOD OF DETERMINING STRUCTURE OF SOFT MATERIAL

Technical Field

The present invention relates to a method of determining structures of low-density materials referred to as soft materials. The term "soft material" as used herein signifies a material whose structure is disorder on an atomic scale but order on a meso-scale (from 20 to 500 angstroms). In particular, the invention is concerned with a method of determining three-dimensional structures of soft materials by use of high-resolution transmission electron microscopy images.

Prior Art

In a traditional structure determination of a material, a sample of the material is extensively irradiated with beams, such as X rays, electron beams or neutron beams, and diffraction patterns (diffraction intensity curves) obtained from the irradiated volume are measured. By such a measurement, the average structure of a sample material in its entirety can be presumed. More specifically, the spatial distribution of scatterers, such as electrons, in unit cells as the smallest structural units is determined on the precondition that atoms are spaced in a completely periodic configuration, and an approximate solution thereto, or the coordinates describing the position of each atom, is determined on the assumption that each atom has a spherically symmetric electron distribution. In this case, the intensity of each diffraction point is

generally measured on an individual basis by the use of a single crystal. From these intensities, the amplitudes of the structure factors are determined, and further the phases of the structure factors are estimated using some method. By inversely Fourier transforming these structure factors, a structure of the sample material can be determined.

In general, the distribution $\rho(x,y,z)$ of a scatterer (atom) concerned with diffraction can be expanded to the following Fourier series when the crystal structure factor is represented as $F(h,k,l)$, the phase factor as $\phi(h,k,l)$ and the scatterer volume as V:

$$\rho(x,y,z)=(1/V)\Sigma(h)\Sigma(k)\Sigma(l)F(h,k,l)\exp\{-2\pi(hx+ky+lz)\} \quad \cdot\cdot(1)$$

$$F(h,k,l)=\mathrm{ABS}\{F(h,k,l)\}\exp\{i\phi(h,k,l)\} \quad \cdot\cdot\cdot(2)$$

wherein h, k and l are indices of diffraction planes.

Accordingly, the structure $\rho(x,y,z)$ can be uniquely determined from inverse Fourier transform so long as the crystal structure factor $F(h,k,l)$, namely the amplitude $\mathrm{ABS}\{F(h,k,l)$ and the phase $\phi(h,k,l)$, is found with respect to a number of hkl reflections.

However, the diffraction means generally adopted for structure determination (such as X-ray diffraction, electron diffraction or neutron-beam diffraction) enables nothing but measurements of diffraction intensities of hkl reflections, or the absolute values of crystal structure factors $\mathrm{ABS}\{F(h,k,l)\}$, but uniquely determination of the phases $\phi(h,k,l)$ cannot be made thereby. Thus, the traditional diffraction means have a drawback that estimations of the phases of the crystal structure factors require a premise that diffraction intensity

measurements have already been made on a great number of diffraction indices.

Further, the traditional diffraction techniques as mentioned above are developed on a basis of periodicity of bonds at an atomic level, so that it is impossible for these techniques to clear up structures of soft materials. This is because, in the diffraction from a soft material structure, several reflection lines are observed in the low scattering-angle region, while in the high scattering-angle region nothing but diffuse scattering is observed; as a result, it is impossible to obtain diffraction intensities for many reflections. For instance, the powder X-ray diffraction pattern of a mesoporous silica SBA-1, is shown in Fig. 4. From this pattern, it is impossible to determine not only a space group but also a crystal system (structural unit cell parameters $a, b, c, \alpha, \beta, \gamma$).

Additionally, the term "soft material" as used herein means a material whose structure is disorder on an atomic scale but in a good order on a meso-scale (from 20 to 500 angstroms).

Problems that the Invention is to solve

As a result of our intensive studies to determine structures of soft materials, it has been found that by taking advantage of low density of a soft material and small dynamic scattering effect of electrons transmitted by a soft material, it becomes possible to uniquely determine the three-dimensional structure of a soft material from high-resolution transmission electron microscopy images, although univocal determination thereof was impossible by the use of the

traditional X-ray or electron diffraction. By this finding, we have achieved the present invention.

Therefore, an object of the invention is to provide a method of easily determining three-dimensional structures of soft materials without making assumptions although it was hitherto difficult to determine them.

Means for Solving the Problems

The aforesaid object of the invention is attained with a method of determining a soft material structure, characterized by comprising steps of taking transmission electron microscopy images of soft materials with crystallographically different directions of incident electrons, Fourier transforming each of the images photographed, evaluating therefrom amplitudes and phases of three-dimensional crystal structure factors, and further performing inverse Fourier transforms by use of the values evaluated, thereby determining a structure of the soft material.

Brief Description of the Drawings

Fig. 1 is a photograph of the high-resolution transmission electron microscopy image taken from a thin film sample of the mesoporous silica SBA-1 with electron beams in the direction [100]. Fig. 2 shows a three-dimensional structure of the mesoporous silica SBA-1 determined by the present method. Fig. 3 shows a three-dimensional structure of the mesoporous silica SBA-16. Fig. 4 shows a powder X-ray pattern (CuKα) of the mesoporous silica SBA-1 in the low scattering-angle region (2θ: 0° to 10°). Additionally, the

characters A and B each in Fig. 2 denote different cavities.

Mode for Carrying Out the Invention

The soft materials relating to the invention, which signify materials whose structures are disorder on an atomic scale but order on a meso-scale (from 20 to 500 angstroms), generally include light elements, porous materials, combinations of light elements, combinations of porous materials and combinations of light elements and porous materials. More specifically, mesoporous materials, surfactants, (copolymerized) macromolecules, biological membranes and liquid crystals are included in the soft materials to which the present invention is applicable.

A transmission electron microscopy image, as is evident from its principle, is a projection of the scatterer atom distribution $\rho(x,y,z)$ viewed from the direction of incident electron beams. In the case of Z-axis incidence, for instance, the transmission electron microscopy image is observed exactly as information concerning the x and y coordinates of the atom distribution integrated with respect to z of $\rho(x,y,z)$. In other words, a group of data for the equation $F(h,k,0) = \mathrm{ABS}\{F(h,k,0)\}\exp\{i\phi(h,k,0)\}$ are determined uniquely on a series of reciprocal lattice points expressed as h,k,l (l=0) by the foregoing equation (1). In analogy with the above case, transmission electron microscopy images are observed respectively from a plurality of directions independent of the above, and subjected to Fourier transform processing to evaluate $F(h,k,l) = \mathrm{ABS}\{F(h,k,l)\}\exp\{i\phi(h,k,l)\}$ with respect to reciprocal lattice points in the three-dimensional

reciprocal space within the limits of resolutions of the images, and then inversely Fourier transformed; as a result, a three-dimensional structure is determined uniquely.

When the foregoing method is carried out, the mean free path of electrons inside a material is generally short because of strong interaction between the electrons and the material, and the electrons are scattered multiply during the propagation through the material sample to produce dynamic scattering effect. Therefore, the structural analysis by such a method has so far been thought to be difficult. However, as the scattering power of a soft material is weak, the dynamic scattering effect as mentioned above becomes negligible when the thickness of a soft material sample is reduced to 50 nm or below. The preparation of such a thin sample can be performed according to known methods. The thinner the sample thickness, the better result is obtained.

In order to determine the three-dimensional structure of a soft material with satisfactorily high accuracy, it is appropriate that a high-resolution transmission microscope be used as the transmission electron microscope in the invention, and it is desirable that at least three different crystallographically significant directions be selected as the incident directions of electrons and transmission electron microscopy images be formed under incidence of electron beams from these directions respectively. The expression "crystallographically significant incident directions of electrons" as used herein means incident-axis directions having high linear independence from one another. For instance,

those incident directions are [100], [110], [111] and [211] in the case of cubic crystals. Needless to say, when transmission electron microscopy images are photographed in more directions of incident electrons and the information derived therefrom is brought into full play, the higher accuracy can be attained in determination of a three-dimensional structure.

In order to perform Fourier transforms of transmission electron microscopy images in the invention, it is required that those images be formed directly on a CCD camera or photographed and then converted into electronic form by means of an image reader. From these data of electronic form, Fourier transform patterns of high-resolution images are obtained in accordance with the usual method. Then, the phases of diffracted waves are read on the assumption of weak topological object approximation. With respect to the diffracted waves in the region of high spatial frequencies, it is desirable that influence of aberration in an objective lens be reduced through estimation of the amount of defocus by the use of a Wiener filter.

In the next place, only peaks on the reciprocal lattice points are selected, and the integral intensity of each peak, from which background is already subtracted in accordance with the method of least squares, is measured. In the case of Fourier diffraction patterns, the phases are calculated simultaneously with the intensity measurement.

When lattice constants are undecided, they are calculated from at least two diffraction patterns obtained in the same field of view and the tilt angle of a sample stage used

in each measurement. Therefrom, several sets of potential lattice constants are derived since the stage angles of now-available electron microscopes are poor in accuracy. When the lattice constants are known, index assignment is carried out for each of the diffraction patterns.

In order to perform structural analysis based on these data, the diffraction data in the TEXT file or the program memory are combined first, and then normalized on the basis of common reflection data. Further, the averaging is carried out by symmetry operation of point groups. At this point, the space group is assumed, and the combined data obtained is stored as a TEXT file.

Fourier diffraction patterns of low spatial resolution (0.3 mm or above) are data of diffraction peaks with phases, while Fourier diffraction patterns of high spatial resolution (0.1 mm or below) are data of diffraction peaks without phases.

Accordingly, each set of these data is read from the TEXT file or the program memory, and phase extension is made by conferring phases on the latter on the basis of the phases of the former.

Then, the diffraction data with phases are read from the TEXT file or the program memory, three-dimensional fast Fourier transform (3D-FFT) thereof is performed to obtain a three-dimensional potential distribution, and peak positions in this distribution are analyzed to assign atom positions.

The invention will now be illustrated in greater detail by reference to the following examples, but these examples should not be construed as limiting the scope of the invention

in any way.

Example 1

Fig. 1 is a high-resolution transmission electron microscopy image obtained by taking a thin film sample of mesoporous silica (SBA-1: Science, 279 (1998) 548-552) under irradiation with electron beams in the direction [100]. This figure shows that the sample has sufficient order on a meso-scale. Particularly in the figure, it is noted that the low-contrast image area corresponding to the periphery of the sample (having a thickness of 50 nm or below) is a region wherein dynamic scattering effect is negligible. The light-and-shade distribution of the image corresponding to the periphery part (50 nm or below in thickness) of the sample was measured with a CCD camera, and the electronic data thus obtained on the light-and-shade distribution were Fourier transformed to evaluate amplitudes and phases of the crystal structure factors. Then, a two-dimensional Fourier diffraction pattern was determined as the distribution of the squared amplitudes. Similarly to the above, transmission electron microscopy images were photographed under irradiation with electron beams incident from the directions [110], [111] and [112] respectively, and therefrom two-dimensional Fourier diffraction patterns were determined.

By the use of all of these diffraction patterns was made a distribution of diffraction intensities on the three-dimensional reciprocal lattice points. From the result obtained, it was determined that the sample has a space group of pm-3n. Further, the origin point of space coordinates was

sought on the basis of the space group determined, and structure factors F(h,k,l) of the three-dimensional reciprocal lattice space were obtained as the phase information and the amplitude information on crystal structure factors. By inversely Fourier transforming those structure factors, the scatterer distribution corresponding to the equation (1) was evaluated, and it was determined that SBA-1 has a structure shown in Fig. 2. More specifically, the structure determined is a structure that voids A and B having different sizes are arranged in amorphous silica in a V3Si configuration (A3B). Additionally, the lattice constant "a" was 73 angstrom.

Example 2

Three-dimensional structures of mesoporous silicas SBA-6 and SBA-16 synthesized under conditions different from that of the mesoporous silica SBA-1, the structures of which had not been elucidated, were each determined using the same method as in Example 1. As a result, it was found that the mesoporous silicas SBA-6 and SBA-1 were identical in structure but different in lattice constant (a=146 angstrom in the case of SBA-6) and sizes of voids A and B. On the other hand, as shown in Fig. 3, the three-dimensional structure of SBA-16 was found to be different from those of SBA-1 and SBA-6, to contain voids which are about 95 angstrom in size and arranged in the form of body-centered cubic lattice, and to have a lattice constant of 133 angstrom.

Claims

1. A method of determining a soft material structure, characterized in that the soft material structure is determined by taking transmission electron microscopy images of a soft material under conditions that a plurality of crystallographically significant directions are selected in succession as incident axes of electrons, Fourier transforming each of the images photographed, evaluating therefrom amplitudes and phases of three-dimensional crystal structure factors, and further performing inverse Fourier transforms by use of the values evaluated.

2. A method of determining a soft material structure as described in claim 1, wherein the transmission electron microscopy images are photographed from at least three different directions.

3. A method of determining a soft material structure as described in claim 1, wherein the soft material is a light element, a porous material, a combination of light elements, a combination of porous materials or a combination of a light element and a porous material.

4. A method of determining a soft material structure as described in claim 1, wherein the soft material is a substance selected from the group consisting of mesoporous materials, surfactants, copolymerized macromolecules, biological membranes and liquid crystals.

5. A method of determining a soft material structure as described in claim 3, wherein the soft material is a substance selected from the group consisting of mesoporous materials,

surfactants, copolymerized macromolecules, biological membranes and liquid crystals.

6. A method of determining a soft material structure as described in claim 1, wherein the images used for Fourier transform are partial areas of images corresponding to no greater than 50 nm-thick parts of a sample of the soft material.

10/031081

JC13 Rec'd PCT/PTO 16 JAN 2002

APPLICATION DATA SHEET

APPLICATION INFORMATION

Application Type::	REGULAR
Subject Matter::	UTILITY
CD-ROM or CD-R?::	NONE
Title::	METHOD OF DETERMINING STRUCTURE OF SOFT MATERIAL
Attorney Docket Number::	TAKIT 163

INVENTOR INFORMATION

Applicant Authority Type::	INVENTOR
Primary Citizenship Country::	JAPAN
Status::	FULL CAPACITY
Given Name::	Osamu
Family Name::	TERASAKI
City of Residence::	Osamu
Country of Residence::	Japan
Street of Mailing Address::	1-7-7, Moniwadai, Taihaku-ku
City of Mailing Address::	Sendai-shi, Miyagi
Country of Mailing Address::	Japan
Postal or Zip Code of Mailing Address::	982-0252

Applicant Authority Type::	INVENTOR
Primary Citizenship Country::	JAPAN
Status::	FULL CAPACITY
Given Name::	Tetsu
Family Name::	OHSUNA
City of Residence::	Sendai-shi, Miyagi
Country of Residence::	Japan
Street of Mailing Address::	2-5-3-703, Itsutsubashi, Aoba-ku
City of Mailing Address::	Sendai-shi, Miyagi
Country of Mailing Address::	Japan
Postal or Zip Code of Mailing Address::	980-0022

DOMESTIC PRIORITY INFORMATION

Application::	Continuity Type::	Parent Application::	Parent Filing Date::
This Application	National Stage of	PCT/JP00/08326	11/27/00

FOREIGN PRIORITY INFORMATION

Application Number::	Country::	Filing Date::	Priority Claimed::
2000-145480	Japan	05/17/00	YES

ASSIGNMENT INFORMATION

Assignee Name::	JAPAN SCIENCE AND TECHNOLOGY CORPORATION
Street of Mailing Address::	4-1-8, Honcho Kawaguchi-shi
City of Mailing Address::	Saitama
Country of Mailing Address::	Japan
Postal or Zip Code of Mailing Address::	351-0198

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY
(Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought of the invention entitled:

METHOD OF DETERMINING STRUCTURE OF SOFT MATERIAL

the specification of which (check only one item below):

☐ is attached hereto.

☐ was filed as United States application

Serial No. ________

on ________

and was amended

on ________ (if applicable).

☒ was filed as PCT international application

Number PCT/JP00/08326

on November 27, 2000,

and was amended under PCT Article 19

on ________ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim priority benefits under Title 35, United States Code, §119 of the following United States Provisional Application and of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR U.S. PROVISIONAL AND FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119
JAPAN	2000-145480	May 17, 2000	☒ YES ☐ NO
			☐ YES ☐ NO
			☐ YES ☐ NO
			☐ YES ☐ NO
			☐ YES ☐ NO

Combined Declaration For Patent Application and Power of Attorney (Continued)

(Includes Reference to PCT, International Applications)

ATTORNEY'S DOCKET NUMBER

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED

PCT APPLICATION NO.	PCT FILING DATE	U.S. SERIAL NUMBERS ASSIGNED (*if any*)			

POWER OF ATTORNEY: As a named inventor, I hereby appoint I. William Millen (19,544); John L. White (17,746); Anthony J. Zelano (27,969); Alan E.J. Branigan (20,565); John R. Moses (24,983); Harry B. Shubin (32,004); Brion P. Heaney (32,542); Richard J. Traverso (30,595); John A. Sopp (33,103); Richard M. Lebovitz (37,067); John H. Thomas (33,460); Catherine M. Joyce (40,668); James T. Moore (35,619), James E. Ruland (37,432), Nancy Axelrod (44,014) and Jennifer J. Branigan (40,921) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

Send Correspondence to: MILLEN, WHITE, ZELANO & BRANIGAN, P.C.
Arlington Courthouse Plaza I, Suite 1400
2200 Clarendon Boulevard
Arlington, Virginia 22201

Telephone No. 703/243-6333

Direct Telephone Calls to:

201	FULL NAME OF INVENTOR	FAMILY NAME TERASAKI	FIRST GIVEN NAME Osamu	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY Sendai-shi, Miyagi JPX	STATE OR FOREIGN COUNTRY Japan	COUNTRY OF CITIZENSHIP Japan
	POST OFFICE ADDRESS	STREET 1-7-7, Moniwadai, Taihaku-ku	CITY Sendai-shi, Miyagi	STATE & ZIP CODE/COUNTRY 982-0252 Japan
202	FULL NAME OF INVENTOR	FAMILY NAME OHSUNA	FIRST GIVEN NAME Tetsu	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY Sendai-shi, Miyagi JPX	STATE OR FOREIGN COUNTRY Japan	COUNTRY OF CITIZENSHIP Japan
	POST OFFICE ADDRESS	STREET 2-5-3-703, Itsutsubashi, Aoba-ku	CITY Sendai-shi, Miyagi	STATE & ZIP CODE/COUNTRY 980-0022 Japan
203	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
204	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY

Combined Declaration for Patent Application and Power of Attorney (Continued)
(Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER

205	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
206	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
207	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
208	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
209	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY
210	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	STREET	CITY	STATE & ZIP CODE/COUNTRY

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

SIGNATURE OF INVENTOR 201 Osamu Terasaki	DATE 17 Nov. 2001 17/11/2001	SIGNATURE OF INVENTOR 207	DATE
SIGNATURE OF INVENTOR 202 Jotan Ohsuna	DATE 17/11/2001	SIGNATURE OF INVENTOR 208	DATE
SIGNATURE OF INVENTOR 203	DATE	SIGNATURE OF INVENTOR 209	DATE
SIGNATURE OF INVENTOR 204	DATE	SIGNATURE OF INVENTOR 210	DATE
SIGNATURE OF INVENTOR 205	DATE	SIGNATURE OF INVENTOR 211	DATE
SIGNATURE OF INVENTOR 206	DATE	SIGNATURE OF INVENTOR 212	DATE